PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-033969
(43)Date of publication of application : 10.02.1998

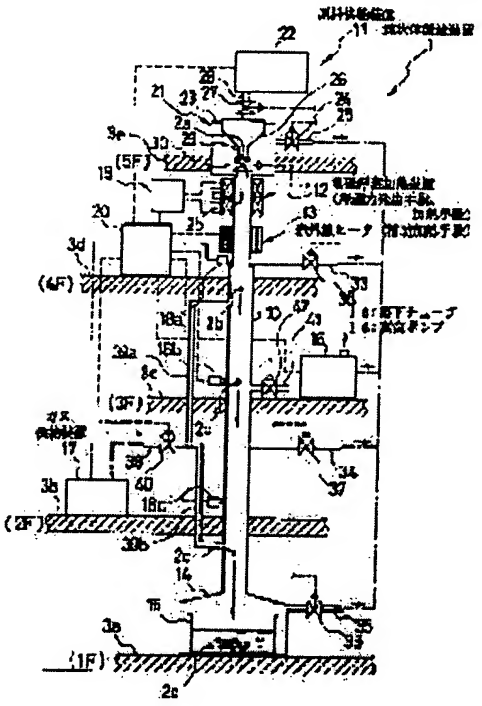
(51)Int.Cl. B01J 2/02
C30B 29/06
C30B 30/08

(21)Application number : 08-211883
(22)Date of filing : 22.07.1996

(71)Applicant : NAKADA JIYOUSUKE
(72)Inventor : NAKADA JIYOUSUKE

(54) MANUFACTURE OF SPHERICAL BODY OF INORGANIC MATERIAL AND MANUFACTURING APPARATUS THEREFOR

(57)Abstract:
PROBLEM TO BE SOLVED: To suppress the formation of projected parts in a part of the surface of a spherical body and lessen the inner strain in the spherical body by releasing heat, heating the surface part of a raw material molten liquid by an auxiliary heating means, and after that cooling the raw material at the time of dropping the raw material molten liquid in a dropping tube.
SOLUTION: When electricity is applied to an electromagnetic suspension heating apparatus 12 and a raw material body 2a is supplied to a dropping tube 10 while opening a shutter 30, the raw material body is suspended in floating state for a short time and heated and becomes a raw material molten liquid 2b. Next, electricity supply to the apparatus 12 is stopped, the raw material molten liquid 2b starts dropping in the tube 10 and is cooled by radiation cooling until is reaches the upper end level of an infrared heater 13 and releases heat. At that time, the raw material molten liquid 2b becomes truly spherical state. After that, only the surface of the raw material molten liquid 2b of the heater 13 is heated and the raw material molten liquid 2b is solidified to be a spherical crystal 2c with a truly spherical shape. Finally, the spherical crystal 2c drops to silicon oil in a silicon oil tank 15 and is completely cooled. In this way, a spherical body in which components are uniformly distributed can be manufactured.



LEGAL STATUS

[Date of request for examination]	17.08.1998
[Date of sending the examiner's decision of rejection]	
[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]	
[Date of final disposal for application]	
[Patent number]	3231244
[Date of registration]	14.09.2001
[Number of appeal against examiner's decision of rejection]	
[Date of requesting appeal against examiner's decision of rejection]	
[Date of extinction of right]	

CLAIMS

[Claim(s)]
[Claim 1] The 1st process to which the raw material object which consists of an inorganic material is heated with a heating means, and carries out melting in the condition of having made it floating in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, the manufacture approach of the spherule made from an inorganic material characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.
[Claim 2] The manufacture approach of the spherule made from an inorganic material according to claim 1 characterized by said inorganic material being silicon.
[Claim 3] The 1st process to which two or more raw material objects which consist of an inorganic material of a mutually different class are heated with a heating means, and carry out melting in one in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, the manufacture approach of the spherule made from an inorganic material characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.
[Claim 4] The manufacture approach of the spherule made from an inorganic material according to claim 3 characterized by the inorganic materials of a class which is different in mutual [said] being silicon and germanium.
[Claim 5] In the equipment which heats the raw material object which consists of an inorganic material in the state of floating in the vacuum in a drop tube, or predetermined gas, is made to solidify the raw material melt, carrying out free fall of the inside of a drop tube, and manufactures a spherule The drop tube of a vertical position, and a raw material object supply means to supply a raw material object from the upper bed into said drop tube, The floating heating means which heats in the condition of having made the raw material object floating in the upper bed section of said drop tube, or its neighborhood, and is made into raw material melt, The manufacturing installation of the spherule made from an inorganic material characterized by having the after heater which is isolated to said floating heating means down side beyond predetermined distance, is arranged in it, and heats the surface section of the raw material melt under drop [inside / of a drop tube].
[Claim 6] The manufacturing installation of the spherule made from an inorganic material according to claim 5 characterized by establishing the vacuation means which makes the inside of said drop tube a vacua through a vacuum pump.
[Claim 7] The manufacturing installation of the spherule made from an inorganic material according to claim 6 characterized by establishing a gas supply means to form the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and to form the gas stream which flows to

the drop direction and opposite direction of a spherule in a drop tube while supplying the predetermined gas according to the class of inorganic material in the drop tube.
[Claim 8] The manufacturing installation of the spherule made from an inorganic material according to claim 7 characterized by preparing the coolant tub which holds in the coolant the spherule which attended the soffit of said drop tube and fell out of the soffit.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.

2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] Especially this invention relates to the technique of manufacturing the spherule made from an inorganic material economically comparatively simply, about the technique of making a spherule solidifying the raw material object of inorganic materials, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass, in an operation of surface tension, carrying out free fall of the inside of an after [melting] drop tube in the state of floating, about the manufacture approach of the spherule made from an inorganic material, and its manufacturing installation.
[0002]
[Description of the Prior Art] Since melting of the inorganic materials various in the bottom of a minute gravity environment is carried out conventionally, the experiment which a spherule is made to solidify has been conducted. The experiment which makes a spherical crystal was also conducted into it. as the melting approach of not using a container for the bottom of a minute gravity environment -- electromagnetism -- the technique which combined synchrotron orbital radiation heating furnaces, such as a halogen lamp, with floating heating, electrostatic floating, or an acoustic wave swimming device has been used. However, since such an experiment was conducted under the minute gravity environment of long duration using the satellite and space shuttle on [instead of the ground] an orbit around the earth, great costs and time amount are taken, and also the constraint on operation is also severe and the count of an experiment is limited. Therefore, it is not suitable for it being restricted to scientific research or an experiment, repeating the spherical crystal of an above-mentioned inorganic material economically for a short time, and producing to a large quantity.
[0003] On the other hand, the minute gravity environment of a short time for 10 or less seconds of it being realizable with the drop tube prepared in the conventional ground and the experimental device of a shot tower method is well-known. for example, in the drop tube type experimental device of U.S. NASA, a drop tube equips on the ground -- having -- the bell jar of the upper bed section -- electromagnetism -- the ** which floating heating apparatus is equipped exchangeable and does not pay the sample of various inorganic materials to a container -- electromagnetism -- floating heating is carried out, and a spherule is made to fuse, and to solidify under minute gravity, carrying out free fall of the inside of the vacuum of a drop tube
[0004] Moreover, the example which made the spherical crystal of silicon using the shot tower is announced ["Development and Evaluation of the Texas Instruments Solar Energy System" 16 th IEEE PVSC Proceedings P.257 -P.260 (1982)]. According to this announcement paper, from the small nozzle with which the upper bed of a shot tower was equipped, the melt of silicon is injected small quantity every, free fall of the inside of the inert gas in a shot tower is carried out, and the spherical crystal of silicon is manufactured. The technique of injecting the melt of silicon small quantity every, carrying out free fall of the inside of a shot tower, and manufacturing the spherical crystal of silicon from the small nozzle with which the U.S. Pat. No. 4,021,323 official report as well as the above was equipped at the upper bed of a shot tower is indicated.
[0005]
[Problem(s) to be Solved by the Invention] As mentioned above, in making the melt of silicon inject from a nozzle and manufacturing the spherical crystal of silicon, it is not suitable for possibility that an impurity will melt into silicon melt from a nozzle being high, and manufacturing the silicon spherical crystal of a high grade. This is also the same as when manufacturing the spherical crystals and spherules made from an inorganic material other than silicon. however, the drop tube type experimental device of NASA -- like --

electromagnetism -- if floating heating apparatus is applied, since silicon melt will not be contacted in a container, an impurity does not melt into silicon melt

[0006] On the other hand, when carrying out free fall of the inside of a vacuum or inert gas and making the melt of silicon solidify, in order to radiate heat from the front face of silicon melt, coagulation begins from the front-face side of silicon melt. However, since a part of silicon melt bulges in the piece place by the side of a front face and a tail-like height is formed when the interior of melt solidifies later than a front-face side, in order to carry out cubical expansion at the time of coagulation, silicon cannot form a real ball-like silicon spherical crystal. In the case of the inorganic material which carries out a volumetric shrinkage at the time of coagulation, a crevice may be formed in the surface section of the spherule solidified reversely with the above. Moreover, if the front-face side of silicon melt solidifies previously, it will become easy to mix the air bubbles adhering to a silicon raw material object in the interior of a spherule.

[0007] And since silicon melt congeals from a front-face side, the internal distortion of the spherical crystal after coagulation also becomes large. When manufacturing the spherical crystal of silicon, annealing processing can remove said internal distortion separately. However, in the case of the inorganic material which cannot carry out the formation postheat treatment of the spherule, it is difficult to remove the internal distortion of a spherule. Anyway, in order to raise the quality of a spherical crystal or a spherule, it is desirable to make internal distortion small as much as possible from the formation phase of a spherical crystal or a spherule.

[0008] In case the object of this invention manufactures the spherule made from an inorganic material, it is offering the manufacture approach of the spherule which can control a tail-like height being formed in a part of front face of a spherule, can make internal distortion of a spherule small, and can prevent mixing of the air bubbles inside a spherule, and its manufacturing installation.

[0009]

[Means for Solving the Problem] The manufacture approach of the spherule made from the inorganic material of claim 1 The 1st process to which the raw material object which consists of an inorganic material is heated with a heating means, and carries out melting in the condition of having made it floating in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.

[0010] as said floating force generating means -- electromagnetism -- floating heating apparatus, an electrostatic swimming device, an acoustic wave swimming device, etc. -- applicable -- as said heating means -- electromagnetism -- floating heating apparatus, an infrared heater, an electric heater, a laser beam heater, a halogen lamp, etc. are applicable. As predetermined gas, oxidizing gases, such as nitrogen gas containing inert gas, such as argon gas, gaseous helium, and nitrogen gas, oxygen gas, or oxygen gas, are applicable. Inorganic materials are which ingredients, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass.

[0011] At the first process [1st], the minute massive raw material object of an inorganic material is supplied to a floating force generating means by a parts feeder etc., by the condition of having made the raw material object floating in a vacuum or predetermined gas with the floating force generating means, it heats with a heating means and melting is carried out. Thus, in order to carry out and change melting of the raw material object into a floating condition, in order that raw material melt may not contact a container, an impurity does not melt into raw material melt. Heat is made to radiate at the 2nd following process with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas.

[0012] In order to carry out free fall of the inside of a drop tube for raw material melt, raw material melt goes into a minute gravity condition, it becomes spherical in an operation of the surface tension of raw material melt, and in order that there may be no effect of gravity or a heat convection, a component serves as melt distributed over homogeneity. When dropping the inside of a vacuum, heat is radiated from the front face of raw material melt by radiation, and when dropping the inside of predetermined gas, heat is radiated from the front face of raw material melt with radiation and heat transfer. By making extent which extent which the coagulation of the front face of said raw material melt does not generate, or partial coagulation does not generate radiate heat, temperature lowering of the whole raw material melt is aimed

at. However, in order to radiate heat from the front-face side of raw material melt, the direction of the surface section becomes low temperature from the interior of raw material melt.
[0013] At the 3rd following process, while raw material melt drops the inside of the vacuum in a drop tube, or predetermined gas, the surface section of raw material melt is heated with an auxiliary heating means. As this auxiliary heating means, in order to heat only the surface section, it is desirable to apply an infrared heater, and it heats so that the temperature of the surface section may become high rather than the temperature inside raw material melt.
[0014] It cools dropping the inside of the vacuum in a drop tube, or predetermined gas, and a spherule is made to solidify raw material melt in an operation of the surface tension of semi-conductor melt in the 4th following process. It solidifies to a spherule, holding the shape of a ball in an operation of surface tension, in order that raw material melt may congeal carrying out free fall. Although raw material melt radiates heat from the surface section at this time, since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section, coagulation begins from both the interior and the surface section from the interior of raw material melt. So, also in the case of the inorganic material which can control effectively that a height is formed in a part of front face of a spherule, and carries out a volumetric shrinkage at the time of coagulation, it can control effectively that a crevice is formed in a part of front face of a spherule, and, also in the case of the inorganic material which carries out cubical expansion at the time of coagulation, internal distortion of a spherule can be made small. Moreover, it is hard coming to mix air bubbles in the spherical inside of the body. And in order to solidify from the condition that there is no seed crystal used as the origin of coagulation initiation, the coagulation in a supercooling condition arises. When an inorganic material is glass, it becomes the spherule of completely new glass by supercooling coagulation.
[0015] Although a part of melt will project in the surface section and a height will be formed in case the interior of raw material melt solidifies if it begins to solidify from the surface section of raw material melt in order to carry out cubical expansion of the silicon especially when an inorganic material is silicon, and solidifying In this invention, even if such a height is not formed and a height is formed, a ********** height to the extent that it disappears separately in the case of annealing processing will only be formed.
[0016] At the 5th following process, a spherule is held in the coolant of the coolant tub which attends the soffit of a drop tube. As this coolant, the liquid (for example, silicone oil) which does not make an impurity melt into a spherule is applied. Thus, by holding the spherule which has fallen in the coolant, a buffer can be aimed at and a spherule can fully be cooled.
[0017] The manufacture approach of the spherule made from the inorganic material of claim 2 is characterized by said inorganic material being silicon in invention of claim 1. It can control that a projection is formed in the surface section of a silicon spherical crystal, and internal distortion of a spherical crystal can be made small as explained in the column of said claim 1.
[0018] The manufacture approach of the spherule made from the inorganic material of claim 3 The 1st process to which two or more raw material objects which consist of an inorganic material of a mutually different class are heated with a heating means, and carry out melting in one in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.
[0019] Although this manufacture approach is the same as that of the manufacture approach of claim 1, and a basic target fundamentally, in the 1st process, it differs at the point which applies two or more raw material objects which consist of an inorganic material of a class which is mutually different, and is the same as that of claim 1 about the 2nd process - the 5th process. That is, in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a floating force generating means, two or more raw material objects which consist of an inorganic material of a class which is mutually different in the 1st process are heated with a heating means, and carry out melting in one. The weight ratio of two or more raw material objects does not restrict that it is the same, but is set up suitably. Since it is the same as that of claim 1 about said inorganic material, a floating force generating means, and a heating means, explanation is omitted. According to this manufacture approach, it is the spherule which consists of an inorganic material with which two or more classes differ, the spherule from which the component was distributed over homogeneity can be manufactured, it can control that a height is formed

in the surface section of a spherule, and internal distortion of a spherule can be made small.
[0020] The manufacture approach of the spherical crystal made from the inorganic material of claim 4 is characterized by the inorganic materials of a class which is different in mutual [said] being silicon and germanium in invention of claim 3. Since silicon and germanium form a complete solid solution, the spherical crystal of the silicon germanium mixed-crystal semiconductor which has a desired mixed-crystal ratio can be manufactured by choosing a presentation ratio as arbitration.
[0021] The manufacturing installation of the spherule made from the inorganic material of claim 5 In the equipment which heats the raw material object which consists of an inorganic material in the state of floating in the vacuum in a drop tube, or predetermined gas, is made to solidify the raw material melt, carrying out free fall of the inside of a drop tube, and manufactures a spherule The drop tube of a vertical position, and a raw material object supply means to supply a raw material object from the upper bed into said drop tube, The floating heating means which heats in the condition of having made the raw material object floating in the upper bed section of said drop tube, or its neighborhood, and is made into raw material melt, It is isolated to said floating heating means down side beyond predetermined distance, is arranged in it, and has the after heater which heats the surface section of the raw material melt under drop [inside / of a drop tube].
[0022] Said inorganic materials are any one ingredient or two or more ingredients, such as a semi-conductor, a superconductor, the magnetic substance, an alloy, and glass, and the raw material object of one kind of ingredient, the raw material object of two or more ingredients, one raw material object, two or more raw material objects, etc. can apply the raw material object of a gestalt variously as a raw material object. About predetermined gas, it is the same as that of claim 1. as said floating heating means -- electromagnetism -- it can apply any of floating heating apparatus, an electrostatic swimming device, heating apparatus and an acoustic wave swimming device, and heating apparatus ** they are, and various heating means, such as an electric heater, an infrared heater, a halogen lamp heater, and a laser beam heater, can be applied as said heating apparatus. As said after heater, various heaters, such as an infrared heater, a halogen lamp heater, a laser beam heater, and an electric heater, are applicable.
[0023] If a feeding means supplies a raw material object from the upper bed into a drop tube, a floating heating means will be heated in the condition of having made the raw material object floating in the upper bed section of a drop tube, or its neighborhood, and will be made into raw material melt. Since raw material melt is in a floating condition, and a container is not contacted, an impurity does not melt into raw material melt. Although raw material melt radiates heat by radiative cooling etc. and the temperature of the whole raw material melt falls while raw material melt falls the inside of a drop tube to the level of an after heater since an after heater is isolated to the floating heating means down side beyond predetermined distance and is arranged in it, the direction of the surface section becomes low temperature from the interior of raw material melt.
[0024] Next, since the surface section of the raw material melt is heated at an after heater, the direction of the surface section becomes an elevated temperature from the interior of raw material melt. Since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section when temperature lowering is carried out to the congealing point, in order to radiate heat from the surface section of raw material melt during drop of raw material melt, after the raw material melt passes an after heater, coagulation begins from both the interior and the surface section from the interior of raw material melt. Consequently, the operation explained to claim 1 and the same operation are done so.
[0025] The manufacturing installation of the spherule made from the inorganic material of claim 6 establishes the vacuation means which makes the inside of said drop tube a vacua through a vacuum pump in invention of claim 5. In case the inside of a drop tube is made into a vacua and a spherule is manufactured, a vacuation means is operated and the inside of a drop tube is made into a vacua.
[0026] In invention of claim 6, the manufacturing installation of the spherule made from the inorganic material of claim 7 establishes a gas supply means to form the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and to form the gas stream which flows to the drop direction and opposite direction of a spherule in a drop tube while supplying the predetermined gas according to the class of inorganic material in a drop tube. For example, when manufacturing a glass spherule and the spherule made from an oxide high-temperature superconductor, a vacuation means will be stopped, the nitrogen gas containing oxidization gas or oxygen gas will be supplied in a drop tube, and a spherule will be manufactured in the gas. In that case, the gas stream which forms the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and flows to the drop direction and opposite direction of a spherule in a drop tube with a gas supply means while supplying gas in a drop tube is formed.
[0027] Although the raw material melt of an about several 100-2000-micrometer minor diameter is

solidified in a **** short time within a drop tube, in order to form the gas stream which flows by this ** mostly in the drop direction of the raw material melt before coagulation, frictional force hardly acts on the raw material melt under coagulation from a gas stream, but raw material melt is solidified in the shape of a real ball. And in order to form the gas stream which flows to the drop direction and opposite direction of a spherule after coagulation, the contact degree of a gas stream and a spherule becomes high, and the reaction of gas and a spherule and cooling of a spherule are promoted.

[0028] In invention of claim 7, the soffit of said drop tube is faced the manufacturing installation of the spherule made from the inorganic material of claim 8, and it prepares the coolant tub which holds in the coolant the spherule which fell out of the soffit. A spherule can be cooled while preventing that a spherule is damaged with an impact, in order to hold the spherule which fell out of the soffit of a drop tube in the coolant of a coolant tub.

[0029]

[Embodiment of the Invention] Hereafter, it explains, referring to a drawing about the gestalt of operation of this invention. The spherule manufacturing installation which first manufactures the spherule made from an inorganic material (the number of diameters 100-2000 micrometers) applied to this invention is explained. As shown in drawing 1 , the spherule manufacturing installation 1 for the diameter of 5-10cm The drop tube 10 of a vertical with a height of about 14m, the electromagnetism arranged on the outside of the upper bed section of the drop tube 10 -- with the floating heating apparatus 12 The infrared heater 13 (auxiliary heating means) as an after heater, and the feeding equipment 11 which supplies one raw material object 2a at a time to the upper bed of the drop tube 10, The silicone oil tub 15 which holds in the hold section 14 which stands in a row in the soffit of the drop tube 10, and attends the soffit of the drop tube 10, The vacuum pump 16 which attracts the air in the drop tube 10, and a gas transfer unit 17, It consists of high speed cameras 18a-18c which photo the pipe line and bulbs, raw material melt 2b under drop [inside / of the drop tube 10], and spherical crystal 2c (spherule), and control unit 20 grade which controls these devices. In addition, the floor [first] - the floor [fifth] floors 3a-3e of works are also illustrated.

[0030] Feeding equipment 11 is equipped with the parts feeder 22 which holds raw material object 2a of the shape of a pellet of the feeder 21 connected to the upper bed of the drop tube 10, and much predetermined sizes, and it supplies at a time to one feeder 21, and the parts feeder 22 is constituted so that degasification of the air of the front face of raw material object 2a may be carried out, while heating raw material object 2a beforehand. the case 23 of a feeder 21 -- electromagnetism -- it connects with a vacuum pump 16 by the siphon 25 which has the closing motion valve 24 -- having -- the acceptance machine 26 within a case 23 -- electromagnetism -- it connects with a parts feeder 22 at the path 28 which has the closing motion shutter 27 -- having -- the outlet path 29 of the acceptance machine 26 -- electromagnetism -- the closing motion shutter 30 is formed and the vacuum within a case 23 is introduced into the acceptance machine 26 through two or more micropores.

[0031] under operation of the spherule manufacturing installation 1 -- electromagnetism -- the closing motion valve 24 is opened and is a vacua in a feeder 21. the case where one raw material object 2a is supplied to a feeder 21 from a parts feeder 22 -- electromagnetism -- the closing motion shutter 30 -- closing and electromagnetism -- the closing motion shutter 27 -- opening -- the inside of the acceptance machine 26 -- raw material object 2a -- supplying -- after that -- electromagnetism -- the closing motion shutter 27 is closed. Supply of feeding equipment 11 is attained to the feeder 21 in one raw material object 2a at every predetermined time (for example, 1 second).

[0032] the siphon 33-35 which attracts the air in the drop tube 10 -- electromagnetism -- the closing motion valves 36-38 are equipped, and these siphon 33-35 is connected to the vacuum pump 16. So that predetermined gas (inert gas, oxidizing gas, etc.) can be passed in the drop tube 10 depending on the class of inorganic material The gas supply line 39 prolonged from a gas transfer unit 17 and its gas transfer unit 17 is formed. branch-pipe 39a which branched from the gas supply line 39 is connected to the upper case section of the drop tube 10, and branch-pipe 39b which branched from the gas supply line 39 is connected to the lower-berth section of the drop tube 10 -- having -- a gas supply line 39 -- electromagnetism -- the closing motion valve 40 is equipped. the middle of the drop tube 10 -- the gas exhaust pipe 41 connected to the section carries out atmospheric-air disconnection -- having -- this gas exhaust pipe 41 -- electromagnetism -- the closing motion valve 42 is equipped.

[0033] Since gas flows by this ** mostly in raw material melt 2b and this direction in the Johan department of the drop tube 10 in supplying predetermined gas from a gas transfer unit 17, frictional force does not act on raw material melt 2b from gas. Since gas flows to spherical crystal 2c and an opposite hand in the bottom half department of the drop tube 10, the contact degree of spherical crystal 2c and gas becomes high. however -- the case where the inside of the drop tube 10 is maintained to a vacuum -- a gas transfer unit 17 -- stopping -- electromagnetism -- the closing motion valves 40 and 42 are closed.

[0034] it is shown in drawing 2 -- as -- electromagnetism -- the floating heating apparatus 12 It consists of high-frequency-current generators 19 (refer to drawing 1) which supply the equal high frequency current of hard flow to the up coil 45, the lower coil 46, and these coils 45 and 46. Line of magnetic force upward with the up coil 45 is generated, and downward line of magnetic force is generated with the lower coil 46. When raw material object 2a consists of inorganic materials of a conductor or a semi-conductor, the induced current occurs in raw material object 2a with the line of magnetic force which changes with high frequency and raw material object 2a is in the mid-position (location of a graphic display) of the up coil 45 and the lower coil 46, Since the upward force of acting on the induced current from line of magnetic force, and the downward force are balanced. raw material object 2a holds a floating condition, and raw material object 2a is heated by the exothermic effect in case the induced current flows the inside of raw material object 2a.
[0035] In this way, if the high frequency current to both the coils 45 and 46 is intercepted after raw material object 2a is heated, fusing and becoming raw material melt 2b, raw material melt 2b will carry out free fall to the direction of the infrared heater 13. By this free fall, raw material melt 2b will be in the minute gravity condition of 10-5G, and will become real ball-like in an operation of surface tension.
[0036] a thing for said infrared heater 13 to heat only a little surface section of raw material melt 2b -- it is -- electromagnetism -- it detaches beyond the predetermined distance set up according to the magnitude and the ingredient of spherule 2c between the floating heating apparatus 12, and it is arranged annularly and attached in the outside of the drop tube 10 possible [fine adjustment of a height location]. This infrared heater 13 has the body 47 of a heater of the shape of a cylinder which consists of infrared radiation ceramics, and can control heating capacity by controlling the current supplied to this body 47 of a heater to a precision. Since raw material melt 2b falls rotating, only the surface section of raw material melt 2b is uniformly heated at the infrared heater 13. in addition -- drawing 2 -- electromagnetism -- the macro-temperature of raw material object 2a heated at the floating heating apparatus 12 and the infrared heater 13 or the whole raw material melt 2b is illustrated, and temperature To is a melting point (congealing point).
[0037] Next, how to apply silicon as an inorganic material, to supply raw material object 2a of silicon using said spherule manufacturing installation 1, and to manufacture spherical crystal 2c of one silicon is explained. first, the first preparation phase -- setting -- electromagnetism -- the closing motion valves 40 and 42 close -- having -- electromagnetism -- the closing motion valves 24, 36-38 -- open -- he and a vacuum pump 16 operate and the inside of the drop tube 10 is made into a predetermined vacua. One raw material object 2a is held in the acceptance machine 26 of feeding equipment 11, and the predetermined current set up beforehand energizes at the infrared neater 13.
[0038] next, electromagnetism -- it energizes to the floating heating apparatus 12 -- having -- electromagnetism -- the closing motion shutter 30 is opened and raw material object 2a supplies in the drop tube 10 -- having -- the raw material object 2a -- electromagnetism -- it holds in the floating condition between predetermined minute time amount with the floating heating apparatus 12, is heated, it fuses, and becomes raw material melt 2b. As the temperature distribution of raw material melt 2b at this time are shown in drawing 3 (a), the interior and the surface section of raw material melt 2b become about 1 appearance.
[0039] next, electromagnetism -- cutoff of the energization to the floating heating apparatus 12 begins to drop raw material melt 2b in the inside of the vacuum of the drop tube 10. At first, the drop rate of raw material melt 2b is a low speed, and the radiative cooling of it is carried out between the minute time amount to which raw material melt 2b falls to the level of the upper bed of the infrared heater 13, and it radiates heat. In order to radiate heat from the surface section of raw material melt 2b at this time, the direction of the surface section becomes low temperature from the interior of raw material melt 2b (refer to temperature distribution of drawing 3 (b)). After this drop initiation, since raw material melt 2b will be in a minute gravity condition, it becomes real ball-like in an operation of the surface tension of raw material melt 2b.
[0040] Next, while falling the interior of the infrared heater 13 of the inside in the drop tube 10, only the surface section of raw material melt 2b is heated, and as the temperature distribution of raw material melt 2b are shown in drawing 3 (c), the direction of the surface section becomes an elevated temperature from the interior of raw material melt 2b. Next, falling under the infrared heater 13, raw material melt 2b radiates heat by radiative cooling, and solidifies the inside of the vacuum of the drop tube 10 to real ball-like spherical crystal 2c in an operation of the surface tension of raw material melt 2b.
[0041] Radiative cooling advances after passing said infrared heater 13, and a continuous line or a two-dot chain line comes to show the temperature distribution of raw material melt 2b in the condition of having carried out temperature lowering to near the congealing-point To to drawing 3 (d). That is, as a result of being cooled by radiative cooling from the surface section, the temperature of the interior of raw

material melt 2b and the surface section becomes about 1 appearance from the temperature-distribution condition shown in drawing 3 (c). Thus, since it solidifies in synchronization from the interior of raw material melt 2b, and surface [both] when raw material melt 2b solidifies from the condition of the temperature distribution shown in drawing 3 (d) as a continuous line, even if raw material melt 2b of silicon carries out cubical expansion during coagulation, a height is not formed in the surface section of spherical crystal 2c, and the internal distortion of spherical crystal 2c also becomes very small. Moreover, when raw material melt 2b solidifies from the condition of the temperature distribution shown in drawing 3 (d) according to a two-dot chain line, in order that coagulation may begin from the interior of raw material melt 2b and the surface section may solidify behind time, a height is not formed in the surface section of spherical crystal 2c, and the internal distortion of spherical crystal 2c also becomes very small.

[0042] Then, spherical crystal 2c in the drop tube 10 which coagulation completed on the level of an inside step mostly falls into the silicone oil in the silicone oil tub 15, is held there, and is cooled thoroughly. Since it is buffered by the silicone oil when falling into a silicone oil, spherical crystal 2c is not damaged. In addition, in much spherical crystal 2c, after manufacture, the closing motion door besides a graphic display is opened, and the silicone oil tub 15 is taken out outside. In addition, although the internal distortion of spherical crystal 2c becomes small as mentioned above, when the whole spherical crystal 2c does not become a single crystal, the whole spherical crystal 2c can be used as a single crystal by carrying out annealing processing separately.

[0043] Even if it expands in case raw material melt 2b of silicon solidifies in order to make spherical crystal 2c solidify according to the spherule manufacturing installation 1 and the spherule manufacture approach which were explained above, after equalizing the temperature distribution of raw material melt 2b like drawing 3 (d) through the infrared heater 13, it can control certainly that a height is formed in the surface section of spherical crystal 2c, and spherical crystal 2c of the shape of a real ball without a height can be manufactured. And even if a height is formed, a height to the extent that it disappears in the case of very small annealing processing will only be formed. In order that the surface section of raw material melt 2b may not solidify beyond the interior, the air bubbles adhering to the front face of raw material object 2a stop moreover, mixing in spherical crystal 2c. Moreover, since raw material melt 2b is solidified under a minute gravity condition and set to spherical crystal 2c, a component is set to spherical crystal 2c distributed over homogeneity, without being influenced of a heat convection, buoyancy, and sedimentation. Moreover, in case raw material melt 2b solidifies, in order to solidify in the condition that there is no seed crystal, it will solidify from a supercooling condition.

[0044] Since the height of said drop tube 10 is about 14m, the drop time amount of raw material melt 2b is for about 1.7 seconds, and can manufacture spherical crystal 2c of the size which can be solidified in these about 1.7 seconds. However, if the die length of the drop tube 10 is enlarged further, much more big spherical crystal 2c can be manufactured. In addition, although the aforementioned example explained as an example the case where spherical crystal 2c of silicon was manufactured, they are not only silicon but various inorganic materials (a semi-conductor, a superconductor, a dielectric, the magnetic substance, an alloy, glass, etc.), and can manufacture the spherical crystal and spherule made from an inorganic material in which induction heating is possible. And also in the case of the inorganic material which carries out a volumetric shrinkage in the case of coagulation, a crevice is not formed in the surface section of spherule 2c, but it becomes a real ball-like spherule. And since feeding equipment 11 is constituted so that raw material object 2a can be supplied into the drop tube 10 every predetermined time, it can mass-produce efficiently the spherical crystal and spherule made from an inorganic material.

[0045] In manufacturing the spherule made from glass or an oxide high-temperature superconductor (for example, YBa2 Cu 3O2) a vacuum pump 16 -- stopping -- electromagnetism -- the closing motion valves 24, 36-38 -- closing and electromagnetism -- the closing motion valves 40 and 42 -- an aperture -- From a gas transfer unit 17, the nitrogen gas containing oxygen gas or oxygen gas is supplied to the drop tube 10, heating melting of the raw material object is carried out in the gas, and the inside of the gas is made to solidify, carrying out free fall of the raw material melt 2b. Moreover, the gas of the inorganic material for doping is supplied into the drop tube 10 with the inert gas supplied from a gas transfer unit 17, and heating melting of the raw material object is carried out in the gas, and you can also make it solidify the inside of the gas, in doping the inorganic material with which classes differ in the surface section of spherical crystal 2c, carrying out free fall of the raw material melt 2b.

[0046] the case of the inorganic material (for example, glass) of an insulator -- electromagnetism -- since induction heating cannot be carried out with the floating heating apparatus 12 -- electromagnetism -- what is necessary is just to apply an electrostatic swimming device, heating apparatus, or an acoustic wave swimming device and heating apparatus instead of the floating heating apparatus 12 In addition, as the heating apparatus, various heating means, such as an electric heater, an infrared heater, a halogen lamp heater, and a laser beam heater, are applicable. On the other hand, various heaters, such as an

electric heater, a halogen lamp heater, and a laser beam heater, are applicable without the class of inorganic material, and relation instead of said infrared heater 13.
[0047] Next, another operation gestalt which changed the aforementioned operation gestalt selectively is explained. it is shown in drawing 4 -- as -- spherule manufacturing installation 1A made from this inorganic material -- two kinds of different raw material objects 2a and 2A made from an inorganic material -- the inside of the drop tube 10 -- supplying -- electromagnetism -- it is equipment which manufactures the spherical crystal which fuses in one with the floating heating apparatus 12, and consists of two kinds of products made from an inorganic material, and a spherule. Since the configuration of those other than feeding equipment 11A is the same as that of the thing of said operation gestalt, it gives the same sign to the same component, and omits explanation.
[0048] feeding equipment 11A -- everything but a parts feeder 22 -- parts feeder 22A -- having -- this parts feeder 22A -- electromagnetism -- it connects with the acceptance machine 26 of a feeder 21 through path 28A which has closing motion shutter 27A, and constitutes also from this parts feeder 22A possible [supply of raw material object 2A] in the acceptance vessel 26. From a parts feeder 22, raw material object 2a of silicon is supplied to the acceptance machine 26, and raw material object 2A of germanium is supplied to the acceptance machine 26 from parts feeder 22A. However, each weight is set up so that raw material object 2a and raw material object 2A may become a predetermined presentation ratio.
[0049] the case where the P type which consists of silicon and germanium, or the spherical crystal of an N type semiconductor is manufactured -- the acceptance machine 26 -- raw material object 2a of one silicon, and raw material object 2A of one germanium -- after hold and said operation gestalt -- the same -- the raw material objects 2a and 2A -- electromagnetism -- it heats in the condition of having supplied the floating heating apparatus 12 and having made it floating in the shape of contact, melting is carried out in one, and it considers as raw material melt. It is the same as that of said operation gestalt after it. Thus, the spherical crystal of the mixed-crystal semiconductor which mixed germanium with silicon can be manufactured. In addition, the spherical crystal and spherule which consist of two kinds of inorganic materials not only among silicon and germanium but among various inorganic materials can be manufactured. In addition, not only two parts feeders 22 and 22A but three or more parts feeders can be prepared, and the spherical crystal and spherule which consist of three or more kinds of inorganic materials can also be manufactured.
[0050]
[Effect of the Invention] a claim -- one -- invention -- depending -- if -- the -- one -- a process -- the -- five -- a process -- a raw material -- the body -- floating -- having made -- a condition -- fusing -- drop -- a tube -- inside -- free fall -- carrying out -- making -- while -- solidifying -- making -- a spherule -- carrying out -- since -- an impurity -- not containing -- a spherule -- a heat convection -- buoyancy -- sedimentation -- effect -- nothing -- a component -- homogeneity -- having been distributed -- an inorganic material -- make -- a spherule (a spherical crystal is included) -- it can manufacture . Moreover, in being able to control effectively that make coagulation start from both the interior and the surface section, and a height and a crevice are formed in a part of front face of a spherule from the interior of raw material melt by making raw material melt solidify through the 2nd process and the 3rd process, internal distortion of a spherule can be made small. Moreover, the air bubbles adhering to the front face of a raw material object can also prevent mixing in the interior of a spherule.
[0051] Since according to invention of claim 2 the same effectiveness as claim 1 is done so and also an inorganic material is silicon, the spherical crystal of silicon can be manufactured, it can control that a height is formed in the surface section of the spherical crystal, it can be used as a real ball-like spherical crystal, and internal distortion of a spherical crystal can be made small.
[0052] According to invention of claim 3, the same effectiveness as claim 1 is done so, and also it is the spherule (a spherical crystal is included) which consists of an inorganic material with which two or more classes differ, the spherule from which the component was distributed over homogeneity can be manufactured, it can control that a height and a crevice are formed in the surface section of a spherule, and internal distortion of a spherule can be made small.
[0053] Although the manufacture approach of the spherical crystal made from the inorganic material of claim 4 does so the same effectiveness as claim 3, since the inorganic materials of a class which is different in mutual [said] are silicon and germanium, it can manufacture the spherical crystal of the mixed-crystal semiconductor of silicon germanium.
 [0054] According to the manufacturing installation of the spherule made from the inorganic material of claim 5, a drop tube, Since it has the raw material object supply means, the floating heating means, and the after heater that is isolated to the floating heating means down side beyond predetermined distance, is arranged in it, and heats the surface section of the raw material melt under drop [inside / of a drop tube], Like claim 1, there is neither a

height nor a crevice in the surface section, by the shape of a real ball, internal distortion is small and the spherule made from an inorganic material without mixing of air bubbles can be mass-produced cheaply efficiently continuously.

[0055] Although the same effectiveness as claim 5 was done so, since the vacuation means which makes the inside of a drop tube a vacua was established according to the manufacturing installation of the spherule made from the inorganic material of claim 6, the inside of a drop tube can be made into a vacua, and a spherule can be manufactured.

[0056] According to the manufacturing installation of the spherule made from the inorganic material of claim 7, do so the same effectiveness as claim 6, but While supplying the predetermined gas according to the class of inorganic material in a drop tube Since a gas supply means to have formed the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and to form the gas stream which flows to the drop direction and opposite direction of a spherule in a drop tube was established When supplying predetermined gas in a drop tube and manufacturing a supply object, frictional force hardly acts on the raw material melt under coagulation from a gas stream, but raw material melt is solidified in the shape of a real ball. And the contact degree of a gas stream and the spherule after coagulation becomes high, and the reaction of gas and a spherule and cooling of a spherule are promoted.

[0057] A spherule can be cooled while preventing that a spherule is damaged with an impact, since the coolant tub which holds in the coolant the spherule which attended the soffit of a drop tube and fell out of the soffit was prepared according to the manufacturing installation of the spherule made from the inorganic material of claim 8, although the same effectiveness as claim 7 was done so.

[Translation done.]

TECHNICAL FIELD

[Field of the Invention] Especially this invention relates to the technique of manufacturing the spherule made from an inorganic material economically comparatively simply, about the technique of making a spherule solidifying the raw material object of inorganic materials, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass, in an operation of surface tension, carrying out free fall of the inside of an after [melting] drop tube in the state of floating, about the manufacture approach of the spherule made from an inorganic material, and its manufacturing installation.

[Translation done.]

PRIOR ART

[Description of the Prior Art] Since melting of the inorganic materials various in the bottom of a minute gravity environment is carried out conventionally, the experiment which a spherule is made to solidify has been conducted. The experiment which makes a spherical crystal was also conducted into it. as the melting approach of not using a container for the bottom of a minute gravity environment -- electromagnetism -- the technique which combined synchrotron orbital radiation heating furnaces, such as a halogen lamp, with floating heating, electrostatic floating, or an acoustic wave swimming device has been used. However, since such an experiment was conducted under the minute gravity environment of long duration using the satellite and space shuttle on [instead of the ground] an orbit around the earth, great costs and time amount are taken, and also the constraint on operation is also severe and the count of an experiment is limited. Therefore, it is not suitable for it being restricted to scientific research or an experiment, repeating the spherical crystal of an above-mentioned inorganic material economically for a short time, and producing to a large quantity.

[0003] On the other hand, the minute gravity environment of a short time for 10 or less seconds of it being realizable with the drop tube prepared in the conventional ground and the experimental device of a shot tower method is well-known. for example, in the drop tube type experimental device of U.S. NASA, a drop tube equips on the ground -- having -- the bell jar of the upper bed section -- electromagnetism -- the ** which floating heating apparatus is equipped exchangeable and does not pay the sample of various inorganic materials to a container -- electromagnetism -- floating heating is carried out, and a spherule is made to fuse, and to solidify under minute gravity, carrying out free fall of the inside of the vacuum of a drop tube

[0004] Moreover, the example which made the spherical crystal of silicon using the shot tower is announced ["Development and Evaluation of the Texas Instruments Solar Energy System" 16 th IEEE PVSC Proceedings P.257 -P.260 (1982)]. According to this announcement paper, from the small nozzle with which the upper bed of a shot tower was equipped, the melt of silicon is injected small quantity every, free fall of the inside of the inert gas in a shot tower is carried out, and the spherical crystal of silicon is manufactured. The technique of injecting the melt of silicon small quantity every, carrying out free fall of the inside of a shot tower, and manufacturing the spherical crystal of silicon from the small nozzle with which the U.S. Pat. No. 4,021,323 official report as well as the above was equipped at the upper bed of a shot tower is indicated.

[Translation done.]

EFFECT OF THE INVENTION

[Effect of the Invention] a claim -- one -- invention -- depending -- if -- the -- one -- a process - the -- five -- a process -- a raw material -- the body -- floating -- having made -- a condition -- fusing -- drop -- a tube -- inside -- free fall -- carrying out -- making -- while -- solidifying -- making -- a spherule -- carrying out -- since -- an impurity -- not containing -- a spherule -- a heat convection -- buoyancy -- sedimentation -- effect -- nothing -- a component -- homogeneity -- having been distributed -- an inorganic material -- make -- a spherule (a spherical crystal is included) - - it can manufacture . Moreover, in being able to control effectively that make coagulation start from both the interior and the surface section, and a height and a crevice are formed in a part of front face of a spherule from the interior of raw material melt by making raw material melt solidify through the 2nd process and the 3rd process, internal distortion of a spherule can be made small. Moreover, the air bubbles adhering to the front face of a raw material object can also prevent mixing in the interior of a spherule.
[0051] Since according to invention of claim 2 the same effectiveness as claim 1 is done so and also an inorganic material is silicon, the spherical crystal of silicon can be manufactured, it can control that a height is formed in the surface section of the spherical crystal, it can be used as a real ball-like spherical crystal, and internal distortion of a spherical crystal can be made small.
[0052] According to invention of claim 3, the same effectiveness as claim 1 is done so, and also it is the spherule (a spherical crystal is included) which consists of an inorganic material with which two or more classes differ, the spherule from which the component was distributed over homogeneity can be manufactured, it can control that a height and a crevice are formed in the surface section of a spherule, and internal distortion of a spherule can be made small.
[0053] Although the manufacture approach of the spherical crystal made from the inorganic material of claim 4 does so the same effectiveness as claim 3, since the inorganic materials of a class which is different in mutual [said] are silicon and germanium, it can manufacture the spherical crystal of the mixed-crystal semiconductor of silicon germanium.
[0054] According to the manufacturing installation of the spherule made from the inorganic material of claim 5, a drop tube, Since it has the raw material object supply means, the floating heating means, and the after heater that is isolated to the floating heating means down side beyond predetermined distance, is arranged in it, and heats the surface section of the raw material melt under drop [inside / of a drop tube], Like claim 1, there is neither a height nor a crevice in the surface section, by the shape of a real ball, internal distortion is small and the spherule made from an inorganic material without mixing of air bubbles can be mass-produced cheaply efficiently continuously.
[0055] Although the same effectiveness as claim 5 was done so, since the vacuation means which makes the inside of a drop tube a vacua was established according to the manufacturing installation of the spherule made from the inorganic material of claim 6, the inside of a drop tube can be made into a vacua, and a spherule can be manufactured.
[0056] According to the manufacturing installation of the spherule made from the inorganic material of claim 7, do so the same effectiveness as claim 6, but While supplying the predetermined gas according to the class of inorganic material in a drop tube Since a gas supply means to have formed the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and to form the gas stream which flows to the drop direction and opposite direction of a spherule in a drop tube was established When supplying predetermined gas in a drop tube and manufacturing a supply object, frictional force hardly acts on the raw material melt under coagulation from a gas stream, but raw material melt is solidified in the shape of a real ball. And the contact degree of a gas stream and the spherule after coagulation becomes high, and the reaction of gas and a spherule and cooling of a spherule are promoted.

[0057] A spherule can be cooled while preventing that a spherule is damaged with an impact, since the coolant tub which holds in the coolant the spherule which attended the soffit of a drop tube and fell out of the soffit was prepared according to the manufacturing installation of the spherule made from the inorganic material of claim 8, although the same effectiveness as claim 7 was done so.

[Translation done.]

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] As mentioned above, in making the melt of silicon inject from a nozzle and manufacturing the spherical crystal of silicon, it is not suitable for possibility that an impurity will melt into silicon melt from a nozzle being high, and manufacturing the silicon spherical crystal of a high grade. This is also the same as when manufacturing the spherical crystals and spherules made from an inorganic material other than silicon. however, the drop tube type experimental device of NASA -- like -- electromagnetism -- if floating heating apparatus is applied, since silicon melt will not be contacted in a container, an impurity does not melt into silicon melt

[0006] On the other hand, when carrying out free fall of the inside of a vacuum or inert gas and making the melt of silicon solidify, in order to radiate heat from the front face of silicon melt, coagulation begins from the front-face side of silicon melt. However, since a part of silicon melt bulges in the piece place by the side of a front face and a tail-like height is formed when the interior of melt solidifies later than a front-face side, in order to carry out cubical expansion at the time of coagulation, silicon cannot form a real ball-like silicon spherical crystal. In the case of the inorganic material which carries out a volumetric shrinkage at the time of coagulation, a crevice may be formed in the surface section of the spherule solidified reversely with the above. Moreover, if the front-face side of silicon melt solidifies previously, it will become easy to mix the air bubbles adhering to a silicon raw material object in the interior of a spherule.

[0007] And since silicon melt congeals from a front-face side, the internal distortion of the spherical crystal after coagulation also becomes large. When manufacturing the spherical crystal of silicon, annealing processing can remove said internal distortion separately. However, in the case of the inorganic material which cannot carry out the formation postheat treatment of the spherule, it is difficult to remove the internal distortion of a spherule. Anyway, in order to raise the quality of a spherical crystal or a spherule, it is desirable to make internal distortion small as much as possible from the formation phase of a spherical crystal or a spherule.

[0008] In case the object of this invention manufactures the spherule made from an inorganic material, it is offering the manufacture approach of the spherule which can control a tail-like height being formed in a part of front face of a spherule, can make internal distortion of a spherule small, and can prevent mixing of the air bubbles inside a spherule, and its manufacturing installation.

[Translation done.]

* NOTICES *

1.This document has been translated by computer. So the translation may not reflect the original precisely.

2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

MEANS

[Means for Solving the Problem] The manufacture approach of the spherule made from the inorganic material of claim 1 The 1st process to which the raw material object which consists of an inorganic material is heated with a heating means, and carries out melting in the condition of having made it floating in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.
[0010] as said floating force generating means -- electromagnetism -- floating heating apparatus, an electrostatic swimming device, an acoustic wave swimming device, etc. -- applicable -- as said heating means -- electromagnetism -- floating heating apparatus, an infrared heater, an electric heater, a laser beam heater, a halogen lamp, etc. are applicable. As predetermined gas, oxidizing gases, such as nitrogen gas containing inert gas, such as argon gas, gaseous helium, and nitrogen gas, oxygen gas, or oxygen gas, are applicable. Inorganic materials are which ingredients, such as a semi-conductor, a superconductor, the magnetic substance, a dielectric, an alloy, and glass.
[0011] At the first process [1st], the minute massive raw material object of an inorganic material is supplied to a floating force generating means by a parts feeder etc., by the condition of having made the raw material object floating in a vacuum or predetermined gas with the floating force generating means, it heats with a heating means and melting is carried out. Thus, in order to carry out and change melting of the raw material object into a floating condition, in order that raw material melt may not contact a container, an impurity does not melt into raw material melt. Heat is made to radiate at the 2nd following process with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas.
[0012] In order to carry out free fall of the inside of a drop tube foı raw material melt, raw material melt goes into a minute gravity condition, it becomes spherical in an operation of the surface tension of raw material melt, and in order that there may be no effect of gravity or a heat convection, a component serves as melt distributed over homogeneity. When dropping the inside of a vacuum, heat is radiated from the front face of raw material melt by radiation, and when dropping the inside of predetermined gas, heat is radiated from the front face of raw material melt with radiation and heat transfer. By making extent which extent which the coagulation of the front face of said raw material melt does not generate, or partial coagulation does not generate radiate heat, temperature lowering of the whole raw material melt is aimed at. However, in order to radiate heat from the front-face side of raw material melt, the direction of the surface section becomes low temperature from the interior of raw material melt.
[0013] At the 3rd following process, while raw material melt drops the inside of the vacuum in a drop tube, or predetermined gas, the surface section of raw material melt is heated with an auxiliary heating means. As this auxiliary heating means, in order to heat only the surface section, it is desirable to apply an infrared heater, and it heats so that the temperature of the surface section may become high rather than the temperature inside raw material melt.
[0014] It cools dropping the inside of the vacuum in a drop tube, or predetermined gas, and a spherule is made to solidify raw material melt in an operation of the surface tension of semi-conductor melt in the 4th following process. It solidifies to a spherule, holding the shape of a ball in an operation of surface tension,

in order that raw material melt may congeal carrying out free fall. Although raw material melt radiates heat from the surface section at this time, since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section, coagulation begins from both the interior and the surface section from the interior of raw material melt. So, also in the case of the inorganic material which can control effectively that a height is formed in a part of front face of a spherule, and carries out a volumetric shrinkage at the time of coagulation, it can control effectively that a crevice is formed in a part of front face of a spherule, and, also in the case of the inorganic material which carries out cubical expansion at the time of coagulation, internal distortion of a spherule can be made small. Moreover, it is hard coming to mix air bubbles in the spherical inside of the body. And in order to solidify from the condition that there is no seed crystal used as the origin of coagulation initiation, the coagulation in a supercooling condition arises. When an inorganic material is glass, it becomes the spherule of completely new glass by supercooling coagulation.

[0015] Although a part of melt will project in the surface section and a height will be formed in case the interior of raw material melt solidifies if it begins to solidify from the surface section of raw material melt in order to carry out cubical expansion of the silicon especially when an inorganic material is silicon, and solidifying In this invention, even if such a height is not formed and a height is formed, a ********** height to the extent that it disappears separately in the case of annealing processing will only be formed.

[0016] At the 5th following process, a spherule is held in the coolant of the coolant tub which attends the soffit of a drop tube. As this coolant, the liquid (for example, silicone oil) which does not make an impurity melt into a spherule is applied. Thus, by holding the spherule which has fallen in the coolant, a buffer can be aimed at and a spherule can fully be cooled.

[0017] The manufacture approach of the spherule made from the inorganic material of claim 2 is characterized by said inorganic material being silicon in invention of claim 1. It can control that a projection is formed in the surface section of a silicon spherical crystal, and internal distortion of a spherical crystal can be made small as explained in the column of said claim 1.

[0018] The manufacture approach of the spherule made from the inorganic material of claim 3 The 1st process to which two or more raw material objects which consist of an inorganic material of a mutually different class are heated with a heating means, and carry out melting in one in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a floating force generating means, Next, the 2nd process made to radiate heat with a melt condition, dropping the inside of the vacuum in the drop tube which made raw material melt the vertical position, or predetermined gas, Next, said raw material melt, dropping the inside of the vacuum in said drop tube, or predetermined gas The 3rd process which heats the surface section of raw material melt with an auxiliary heating means, and the 4th process which it cools [process] next, dropping the inside of the vacuum in said drop tube, or predetermined gas, and makes it solidify said raw material melt to a spherule in an operation of the surface tension of raw material melt, Next, it is characterized by having the 5th process which holds said spherule in the coolant of the coolant tub which attends the soffit of said drop tube.

[0019] Although this manufacture approach is the same as that of the manufacture approach of claim 1, and a basic target fundamentally, in the 1st process, it differs at the point which applies two or more raw material objects which consist of an inorganic material of a class which is mutually different, and is the same as that of claim 1 about the 2nd process - the 5th process. That is, in the condition of having made it floating in the shape of contact in a vacuum or predetermined gas with a floating force generating means, two or more raw material objects which consist of an inorganic material of a class which is mutually different in the 1st process are heated with a heating means, and carry out melting in one. The weight ratio of two or more raw material objects does not restrict that it is the same, but is set up suitably. Since it is the same as that of claim 1 about said inorganic material, a floating force generating means, and a heating means, explanation is omitted. According to this manufacture approach, it is the spherule which consists of an inorganic material with which two or more classes differ, the spherule from which the component was distributed over homogeneity can be manufactured, it can control that a height is formed in the surface section of a spherule, and internal distortion of a spherule can be made small.

[0020] The manufacture approach of the spherical crystal made from the inorganic material of claim 4 is characterized by the inorganic materials of a class which is different in mutual [said] being silicon and germanium in invention of claim 3. Since silicon and germanium form a complete solid solution, the spherical crystal of the silicon germanium mixed-crystal semiconductor which has a desired mixed-crystal ratio can be manufactured by choosing a presentation ratio as arbitration.

[0021] The manufacturing installation of the spherule made from the inorganic material of claim 5 In the equipment which heats the raw material object which consists of an inorganic material in the state of floating in the vacuum in a drop tube, or predetermined gas, is made to solidify the raw material melt, carrying out free fall of the inside of a drop tube, and manufactures a spherule The drop tube of a vertical

position, and a raw material object supply means to supply a raw material object from the upper bed into said drop tube. The floating heating means which heats in the condition of having made the raw material object floating in the upper bed section of said drop tube, or its neighborhood, and is made into raw material melt. It is isolated to said floating heating means down side beyond predetermined distance, is arranged in it, and has the after heater which heats the surface section of the raw material melt under drop [inside / of a drop tube].

[0022] Said inorganic materials are any one ingredient or two or more ingredients, such as a semiconductor, a superconductor, the magnetic substance, an alloy, and glass, and the raw material object of one kind of ingredient, the raw material object of two or more ingredients, one raw material object, two or more raw material objects, etc. can apply the raw material object of a gestalt variously as a raw material object. About predetermined gas, it is the same as that of claim 1. as said floating heating means -- electromagnetism -- it can apply any of floating heating apparatus, an electrostatic swimming device, heating apparatus and an acoustic wave swimming device, and heating apparatus ** they are, and various heating means, such as an electric heater, an infrared heater, a halogen lamp heater, and a laser beam heater, can be applied as said heating apparatus. As said after heater, various heaters, such as an infrared heater, a halogen lamp heater, a laser beam heater, and an electric heater, are applicable.

[0023] If a feeding means supplies a raw material object from the upper bed into a drop tube, a floating heating means will be heated in the condition of having made the raw material object floating in the upper bed section of a drop tube, or its neighborhood, and will be made into raw material melt. Since raw material melt is in a floating condition, and a container is not contacted, an impurity does not melt into raw material melt. Although raw material melt radiates heat by radiative cooling etc. and the temperature of the whole raw material melt falls while raw material melt falls the inside of a drop tube to the level of an after heater since an after heater is isolated to the floating heating means down side beyond predetermined distance and is arranged in it, the direction of the surface section becomes low temperature from the interior of raw material melt.

[0024] Next, since the surface section of the raw material melt is heated at an after heater, the direction of the surface section becomes an elevated temperature from the interior of raw material melt. Since the temperature inside raw material melt is lower on a par with the temperature of the surface section than the temperature of the surface section when temperature lowering is carried out to the congealing point, in order to radiate heat from the surface section of raw material melt during drop of raw material melt, after the raw material melt passes an after heater, coagulation begins from both the interior and the surface section from the interior of raw material melt. Consequently, the operation explained to claim 1 and the same operation are done so.

[0025] The manufacturing installation of the spherule made from the inorganic material of claim 6 establishes the vacuation means which makes the inside of said drop tube a vacua through a vacuum pump in invention of claim 5. In case the inside of a drop tube is made into a vacua and a spherule is manufactured, a vacuation means is operated and the inside of a drop tube is made into a vacua.

[0026] In invention of claim 6, the manufacturing installation of the spherule made from the inorganic material of claim 7 establishes a gas supply means to form the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and to form the gas stream which flows to the drop direction and opposite direction of a spherule in a drop tube while supplying the predetermined gas according to the class of inorganic material in a drop tube. For example, when manufacturing a glass spherule and the spherule made from an oxide high-temperature superconductor, a vacuation means will be stopped, the nitrogen gas containing oxidization gas or oxygen gas will be supplied in a drop tube, and a spherule will be manufactured in the gas. In that case, the gas stream which forms the gas stream which flows by this ** mostly in the drop direction of raw material melt in a drop tube, and flows to the drop direction and opposite direction of a spherule in a drop tube with a gas supply means while supplying gas in a drop tube is formed.

[0027] Although the raw material melt of an about several 100-2000-micrometer minor diameter is solidified in a **** short time within a drop tube, in order to form the gas stream which flows by this ** mostly in the drop direction of the raw material melt before coagulation, frictional force hardly acts on the raw material melt under coagulation from a gas stream, but raw material melt is solidified in the shape of a real ball. And in order to form the gas stream which flows to the drop direction and opposite direction of a spherule after coagulation, the contact degree of a gas stream and a spherule becomes high, and the reaction of gas and a spherule and cooling of a spherule are promoted.

[0028] In invention of claim 7, the soffit of said drop tube is faced the manufacturing installation of the spherule made from the inorganic material of claim 8, and it prepares the coolant tub which holds in the coolant the spherule which fell out of the soffit. A spherule can be cooled while preventing that a spherule is damaged with an impact, in order to hold the spherule which fell out of the soffit of a drop tube in the

coolant of a coolant tub.
[0029]
[Embodiment of the Invention] Hereafter, it explains, referring to a drawing about the gestalt of operation of this invention. The spherule manufacturing installation which first manufactures the spherule made from an inorganic material (the number of diameters 100-2000 micrometers) applied to this invention is explained. the electromagnetism which has arranged the spherule manufacturing installation 1 for the diameter of 5-10cm on the outside of the upper bed section of the drop tube 10 of a vertical with a height of about 14m, and the drop tube 10 as shown in drawing 1 -- the floating heating apparatus 12 and the infrared heater 13 as an after heater

---

[Translation done.]

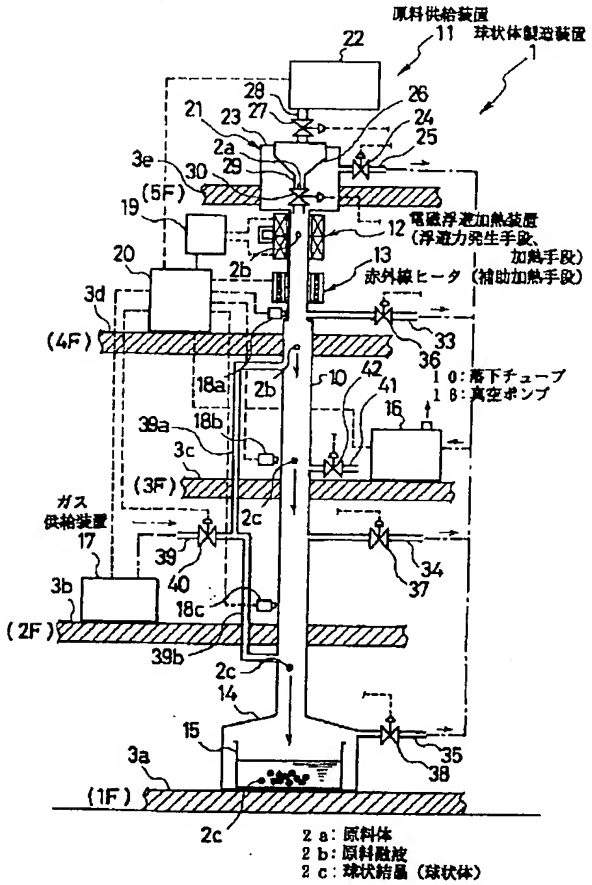
DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the whole spherule manufacturing installation block diagram made from an inorganic material concerning the operation gestalt of this invention.
[Drawing 2] the electromagnetism of said manufacturing installation -- it is the block diagram of floating heating apparatus and an infrared heater.
[Drawing 3] (a) -- electromagnetism -- the thermal mapping of the raw material melt after floating heating and (b) are [the thermal mapping of the raw material melt after heating and (d of the thermal mapping of the raw material melt in front of an infrared heater and (c))] the thermal mapping of the raw material melt in front of coagulation at an infrared heater.
[Drawing 4] It is the whole spherule manufacturing installation block diagram made from an inorganic material concerning another operation gestalt.
[Description of Notations]
1 1A Spherule manufacturing installation
2a, 2A Raw material object
2b Raw material melt
2c Spherical crystal (spherule)
10 Drop Tube
11 Feeding Equipment
12 Electromagnetism -- Floating Heating Apparatus (Floating Force Generating Means, Heating Means, Floating Heating Means)
13 Infrared Heater (Auxiliary Heating Means, after Heater)
16 Vacuum Pump
17 Gas Transfer Unit

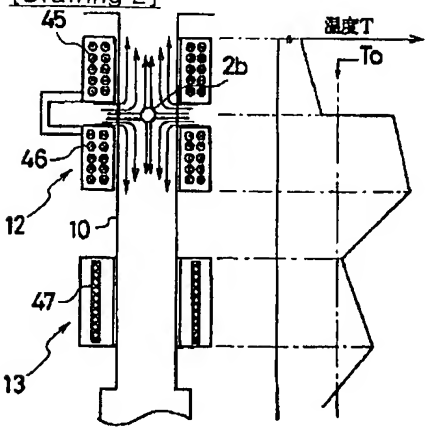
[Translation done.]

DRAWINGS

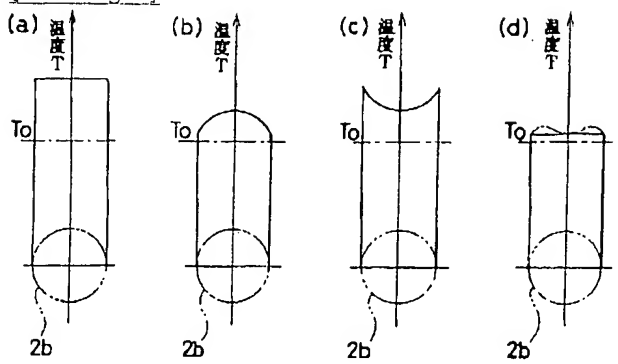
[Drawing 1]



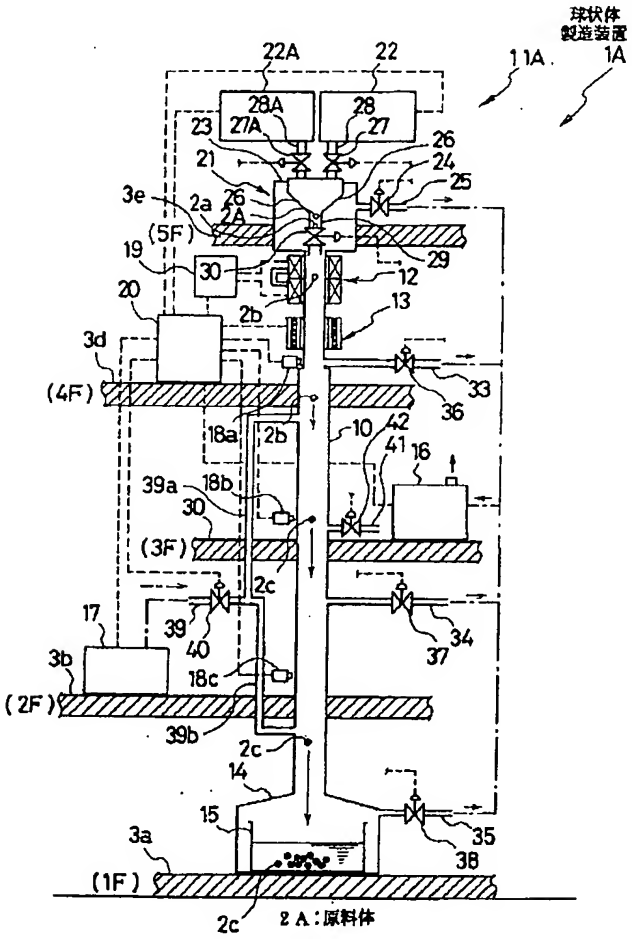
[Drawing 2]



[Drawing 3]



[Drawing 4]



[Translation done.]